

US Patent Application 09/880556  
**BIOCOTE & DEALINGS WITH DUPONT**  
**Inhibition of Bacterial Growth**

Your Ref: S1011/20122

LS&A Ref: P03111US

1. **AFFIDAVIT – SIDNEY THOMAS HARRIS**
2. **NUOSEPT S**
3. **FUNGITROL**
4. **CANGUARD**
5. **SAMPLE 1A**
6. **SAMPLE 1B**
7. **SAMPLE 2**
8. **SAMPLE 3A**
9. **SAMPLE 3B**
10. **SAMPLE A**
11. **SAMPLE B**
12. **JP 06-025561**
13. **JP 08-06036**

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IN THE MATTER OF  
US patent application  
NO. 09/880556  
dated June 13, 2001  
in the name of  
BRODIE and CLOVER

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**AFFIDAVIT**

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I, **SIDNEY THOMAS HARRIS**, a British subject of The Old Rectory, Tamworth Road, Nether Whitacre, Warwickshire, England B46 2DY do hereby state under Oath as follows:

1. I am a qualified chemist experienced in the field of polymers having worked in several industrial organisations. I have spent more than fifty eight years in the coatings industry and have accumulated research and technical development experience in all aspects of the coatings industry. Since the mid 1960's I have been actively engaged with the development of powder grade resin systems and powder coatings formulation. During that period I commissioned more than twenty powder coating manufacturing units world-wide and four resin plant installations for the manufacture of solid grade epoxy resins and a wide range of polyester resins. I am the author of the first authoritative textbook on the subject of powder coatings entitled "*The Technology of Powder Coatings*" (1976). I am a chemistry graduate of London University and have presented more than one hundred technical papers at conferences throughout the world and established joint venture

manufacturing in China and India. In 2002, I installed a powder coating manufacturing unit in Pakistan. I have contributed innumerable articles to trade journals on all aspects of the powder coatings industry. As an independent consultant I am retained in this capacity by several key players in the industry and have successfully appeared as a technical expert in several United Kingdom High Court cases.

2. I am the editor of the monthly bulletin "*Focus on Powder Coatings*" published by Elsevier and contribute a regular monthly column in the Paint & Polymer Coatings Journal. In January 2003, I was retained as a consultant by Oxyplast Belgium N V. (Oxyplast are one of the major established producers of powder coatings with licensees in 23 countries world-wide). I am currently working with RAN Chemicals of Nagpur, India in the development of modern binder resins for powder coatings.
3. I act as technical consultant to BioCote Ltd, the owner of record of the above patent application. I have done so since September 1996.
4. I verily believe that by virtue of my academic and industrial experience over many years, I am competent to comment on the issues in the Official Action mailed September 20, 2002.
5. I have read the text of US patent application 09/880556 and the Official action of September 20, 2002 and cited references.
6. I have read the proposed amendments to the Claims to deal with the objections in paragraphs 4, 5 and 6 of the Official Action. I understand the

language of these Claims in amended form and I shall proceed on the basis that these are the Claims in prosecution.

7. I draw attention to the fact that the following are known solid biocides:

3,5-dimethyltetrahydro-1,3,5-2H-thiazine-2-thione which is available commercially as NUOSEPT S – see attached literature.

N-(trichloromethyl)-thiophthalamide which is available commercially as FUNGITROL – see attached literature.

2-bromo-2-nitropropane-1,3-diol which is available commercially as CANGUARD – see attached literature.

Because these are well known solid biocides and the passage at column 1, lines 48 onwards of the patent specification tells me that a man skilled in the art can determine which to use I say that to such a person the use of these materials and the proportions is obvious and within the disclosure of the specification.

8. I note that the examiner objects to certain Claims because he says that the reader cannot find the parallel wording in the description of the patent. The Claims which relate to a liquid biocide are:

*Claim 19 which reads:*

A powder coating composition of Claim 1, wherein the biocide comprises a liquid biocide.

*Claim 20 which reads:*

The composition of Claim 10, wherein the anti-microbial agent comprises a liquid anti-microbial agent.

- 9.. A powder composition can be made in a variety of ways, one of which involves the use of extruders. A screw extruder can take a variety of forms, one of which is a long cylinder or barrel inside which there is a rotating length of rod having vanes called the screw and the purpose of which is to mix the ingredients as they travel through the barrel. In some cases the barrel has inlet ports in the barrel wall from which liquid can be injected into the mix. I know from my experience in the powder coating industry that where one wants an even distribution of biocide, especially in a low concentration, it is a better practice to mix the liquid first with a solid powder and then mix that powder with the polymer ingredients. The powder acts as a carrier and will assist in the mixing and the dispersion of the liquid. The carrier may be inert or may be of a chemical composition which will also have a benefit in terms of the characteristics, for example, a fluidity additive. For these reasons I say that I understand, as would one skilled in the art in June 1996, from the patent specification that the invention envisage both solid and liquid biocides and as one skilled in the art in June 1966, I would have no problem in understanding how to use the option of a liquid biocide. In this context I refer in particular to the passage from column 1, lines 48 onwards:

"Many biocides may be suitably employed in the invention and the average skilled man of the art will readily be able to determine by routine experimentation whether the biocidal activity of any particular biocide will be sufficiently retained for his particular needs in the

coating. The necessary criteria are that the biocide can be provided in a suitable powder form and that it can survive the coating process.”

I say that this passage tells me and one skilled in the art at the time this application was filed that any solid or liquid biocide can be used, and in the latter case is one which can be embodied in a carrier. Therefore I say that the specification of the instant application discloses the subject matter of Claims 19, 20, 25, 26 and 27.

10. I note also that the examiner is of the view that this present invention is the same as that shown in three earlier documents:

US 5238749 (Cueman)

Japan 06-02251

Japan 08-060036

11. I am familiar with US 5238749, but I cannot remember when I first considered it. For the purposes of the present patent application, I carried out the following tests under proper scientific procedures (all parts are by weight).

12. **Experimental:**

Clear Nylon 11 powder of very fine particle size was dry blended with 2% and 5% of Triclosan in accordance with the preferred concentration range of the Cueman patent, US patent no. 5238749.

SAMPLE 1A was based on clear Nylon 11 containing 2% Triclosan. The components were mixed in a coffee grinder and sprayed onto aluminium panels (so called Q panels) using a corona charging electrostatic powder gun.

Visual inspection prior to stoving showed random scattering of Triclosan and complete absence of uniformity. After stoving at 220°C for 10 minutes, the Triclosan particles had melted and formed clusters at isolated spots in the film. (Stoving is the technical term for the heating in this context.)

SAMPLE 1B was based on clear Nylon 11 containing 5% Triclosan. The components were mixed in a coffee grinder and sprayed onto aluminium Q panels using a corona charged electrostatic powder gun. Visual inspection prior to stoving showed a more intense but random scattering of the Triclosan and again, complete absence of uniformity. After stoving for 10 minutes at 220°C, melted Triclosan was again apparent in clusters but in isolated areas amounting to about 5% in total of the surface area of the protective coating.

SAMPLE 2 – TP2 Unmodified clear Nylon 11 (100%) was applied as a control. The stoved film was clear with no visible defects.

SAMPLE 3A was a pigmented epoxy powder coating containing 2% Triclosan and dispersed in a coffee grinder. The blended powder was applied to aluminium Q panels using the charged corona gun as before to provide a base coat. This film was then overcoated with the Nylon 11 blend TP1 of Sample 1A using a positively charged tribo-electric gun. Visual inspection showed random clusters of Triclosan. The panels were then stoved at 220°C for 10 minutes. There was visual evidence of the heterogeneous scattering of Triclosan in the stoved film.

SAMPLE 3B – the same pigmented epoxy powder coating as in Sample 3A was mixed with 5% of Triclosan and applied in the same manner. It was then

over coated with the modified Nylon 11 of TP2 by tribo-application. The same visual faults were observed prior to and after stoving.

Attached as Exhibits are photographs and samples of the above results.

13. To demonstrate the efficacy of the instant invention two tests were carried out using unpigmented resins.

The dry components of an uncured epoxy-polyester or a polyester coating compositions were separately made up to contain 3% by weight of Triclosan. Each composition was mixed, and the mixture fed to an extruder and heated to a suitable melting temperature, cooled, broken into pieces and granulated. The powder was sprayed onto test panels using a corona charging electrostatic powder gun. The results are shown in **Exhibits A and B** from which it can be seen that the coating is uniform and there are no areas free of biocide. I attribute this effect to the fact that the particles of biocide were homogeneously distributed through the powder particles because of the way in which the compositions were prepared.

14. I conclude that:

- 1) the method described in the Cueman patent, US 5238749, does not give a homogeneous dispersion of biocide in the stoved film; and
- 2) this lack of homogeneity and uniformity of biocide dispersion will make the applied film incapable of providing a reliable defence against microbial attack.



15. I note that the invention of the instant patent application is based on the fact that the biocide is homogeneously distributed through the powder – see column 2, lines 28 onwards. I can see nothing in the Cueman patent which tells me to do this or suggests one to do so.
16. I note that in the specification of the instant patent application, at column 2, lines 28 onwards.

“As each particulate contains the biocide it is homogeneously distributed throughout the coating composition and so cannot separate out. Thus, for example, such a powder may be made by adding the biocide at the initial mixing stage of the constituents which are to be converted into the desired matrix powder. In a typical powder manufacturing process, the precursors of the desired powder, e.g. the resin base and the hardener, together with any additives, are mixed, heated and extruded to sheet form, the sheet is granulated and then ground to the desired powder size.”

This tells me one way how to add the biocide. If I, or one skilled in the art, had read the specification when it was lodged in June 1996 I, or the person skilled in the art, would have understood how to reproduce the invention.

17. With the benefit of the experience I have had working on BioCote projects I can say that the invention of the instant patent application works because each particle of plastic contains a particle of the biocide. Particles of plastics and biocide may have a different capacity to accept an electrostatic charge. Particles of different density behave differently when electrostatically sprayed, and because of this feature there is less risk that the biocide particles (which

differ in density from the plastics one) will reach the metal surface at different rates. As a result the surface will have no biocide in some areas of the metal and a high concentration elsewhere. Nothing in Cueman tells me or one skilled in the art, of this aspect nor does he suggest such a thing. For this reason I say that if I, or one skilled in the art, had seen Cueman before the date i.e. June 5, 1996, it would not have shown or suggested to me or one skilled in the art, the BioCote invention as defined in the Claims.

18. I have read the machine-produced translation of JP 06-025561 published in February 1994. This document teaches the incorporation of defined metal salts in a powder coating composition. When one reads the instructions, see the Examples for instance, they just talk of blending, i.e. they do not promise or achieve a homogenous distribution. I say that this document is not relevant to the invention of the present patent application.
19. I now turn to consider the machine-produced translation of JP 08-060036 which was published in March 1996. It proposes adding metal salts, e.g. salts of silver copper and zinc the ions which are known to be antibacterial, to a powder coating composition. The metal salts are not added directly but are carried within a matrix of a defined zeolite. Paragraph [0019] points out that the presence of the antifungal agent can affect the reaction of the polyester resin and the curing agent therefor, and that the fact that the metal salt is carried on the zeolite avoids this problem.

The first thing to note is that for a metal anti bacterial agent to be carried in the lattice of a zeolite, it must ionise in solution otherwise the agent cannot reach into the lattice. This limits the usefulness of the invention of JP 06-

225561 to anti bacterial **metal** agents. The plastics disclosed in the Abstract is a thermoplastic, i.e. PVC or Nylon.

In contrast, the agents in the instant invention are all organic anti bacterial agents. This invention is concerned with the carrying of such agents into a thermosetting polymer composition, irrespective of whether or not those agents will ionise in contact with water. I say that this document is not relevant to the invention of this instant patent application.

I, **SIDNEY THOMAS HARRIS**, hereby declare that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that wilful, false statement and the like so made are punishable by fine, or imprisonment or both, under Section 1001 of Title 18 of the United States Code, and that such wilful, false statements may jeopardize the validity of this application or any patent issuing therefrom.

SWORN by the said  
SIDNEY THOMAS HARRIS  
at Birmingham, England  
this 26th day of February  
2003 before me,

*Anthony Villiers Blakemore*  
Notary Public  
Birmingham, England.

Further Declareth sayeth not:

*S.T. Harris*  
SIDNEY THOMAS HARRIS

*26th February 2003*  
DATED

LS/BW/vms/26.02.03  
P03111US.HARRIS.AFF.1

MY FACULTY  
ENDURES SO LONG  
AS I PRACTISE  
ANTHONY VILLIERS BLAKEMORE